# Center Reflections

A monthly publication highlighting activities at the W.M. Keck Foundation Center for Molecular Structure

California State University Fullerton

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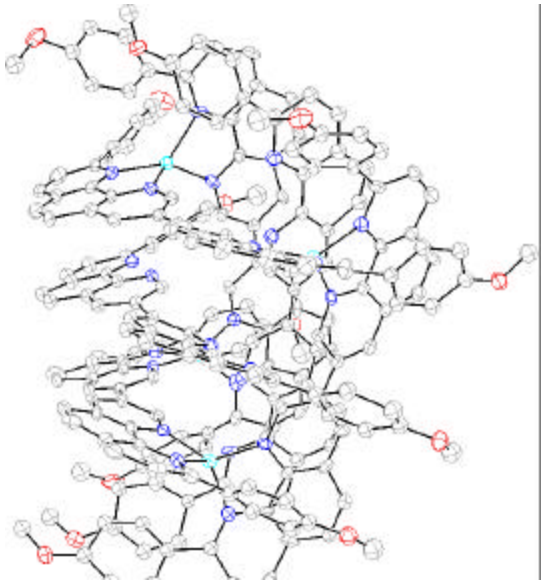
## Kenneth Hardcastle's Copper Chelate Complexes

### CSU Northridge

At California State University Northridge, Professor Kenneth Hardcastle collaborates with other synthetic chemistry groups around the world: Ed Rosenberg at the University of Montana, Paolo Finocchairo in Catania, Italy, the NMR group at the University of Torino, Italy, and Jay Siegel of the Department of Chemistry, UCSD. Jay is a former chemistry graduate from CSUN, who did his PhD degree with Professor Misloe at Princeton. Jay and Kenneth have been collaborating on extended ring, organic structures, such as substituted corannulenes and metal complexes of aromatic heterocycles with the aim of examining in some detail, bond alternation in aromatic structures and investigating the so-called Mills-Nixon effect. Many of the compounds are host-guest type molecules, and the structures seem to grow larger, the longer the collaboration continues.

This collaboration resulted, recently, in the most difficult structure Kenneth has yet attempted, that of a CuGrid complex, a complex formed from ligands of bis(2-anisole-1,10-phenthroline) and Cu(I) ions. Crystals of the CuGrid, obtained from dichloromethane/hexane solution were nicely faceted, but extremely small. Numerous attempts were made to collect data on the crystals over a year, even synchrotron data, with little or no success. A reasonable quality, high-resolution data set was obtained with the HISTAR system at CMolS. The structure has been refined to an R=0.15 for 31,421 reflections and 1756 parameters. The cell has parameters of 17.5893(1), 40.3378(4), 30.5005(4) and β=94.590(1), with a cell volumn of 21,571$A^3$, monoclinic space group $P2_1/n$. This most interesting and complex inorganic macrocyle is shown on the next page.



Dr. Hardcastle has been a professor at CSUN since 1963. He received his AB from San Jose State University and his PhD from USC. In 1994, the Northridge earthquake damaged much of the chemistry department at CSUN, including Hardcastle's CAD4 diffractometer. While he brought his instrument back into working order, he became director of the x-ray crystallography laboratory at Caltech. His former graduate student, Michael Day, currently holds this position.

## Identifying Crystalline Inclusions in Ascidians

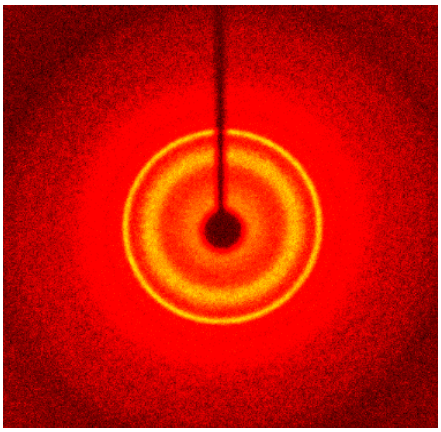
### CSU Fullerton

Professor Katherine Kantardjieff is collaborating with Professor Charles and Gretchen Lambert to identify crystalline inclusions in the body walls of ascidians. It has long been known that a number of ascidians sequester urates and other purines in their bodies over long periods of time. (Goodbody, '65).[1] These appear as scattered microcrystalline inclusions that may be identified by a combination of x-ray powder diffraction, spectroscopy and biochemical assay. We have identified microcrystals of uric acid in the ascidian, *Corella inflata*, by x-ray powder diffraction (shown below), the characteristic UV absoprtion for urates at 292nm which is abolished by uricase, and by methenamine-silver staining in situ.[2] Sadia Khan, a senior research student in Kantardjieff's laboratory, is currently examining microcrystalline inclusions from the body wall of *Ascidia callosa* by powder x-ray diffraction.[3]


1. Goodbody, I. (1965) Nitrogen excretion in Ascidiacea. II. Storage excretion and the uriolytic enzyme system. J. Exp. Biol., 42: 299-305.

2. Charles C. Lambert, Gretchen Lambert, Guy Crundwell and Katherine A. Kantardjieff. (1998). "Uric acid accumulation in the solitary ascidian *Corella inflata*." J. Exper. Zool. 282:323-331.

3. In Program Abstracts, ACS National Meeting, Anaheim, CA, March 20-26, 1999.

## "Crystallography for Chemists" Workshop Given to CSU Faculty in June 1998

In June 1998, CMolS hosted a workshop, "Crystallography for Chemists", which was attended by one dozen CSU

faculty representing six CSU campuses. The objective of this two-day workshop is to provide an easy-to-understand introduction to the use of single-crystal analysis as an analytical tool and to help faculty effectively interact with CMolS. Workshop leaders included Prof. Alex McPherson of UC Irvine and Dr. Charles Campana of Bruker Analytical X-ray Systems. Campana and Kantardjieff have since developed this workshop into a two-day short course sponsored by the American Chemical Society[1], intended for chemists, biochemists, materials scientists and technicians whose research requires structural information, but who have little or no background in x-ray crystallography. Kantardjieff has been invited to give this course at the University of Vienna in May 1999.

1. Kantardjieff, Katherine A. and Campana, Charles F. (1999) Crystallography for Chemists - A Two-Day Short-Course. In program Abstracts, American Crystallographic Association, Annual meeting, Buffalo, NY.

"**Crystallography for Chemists" Participants** - Front Row (L-R): Ray Trautman (SFSU); John Tate (CSUSB); Kenneth Hardcastle (CSUN); Katherine Kantardjieff (CMolS); Charles Campana (Bruker-AXS). Back Row (L-R): Guy Crundwell (CMolS); Charles Bowen (Cal Poly Pomona); Alex McPherson (UCI).

# Upcoming Events

March 14-17, 1999: **West Coast Protein Crystallography Workshop**, Asilomar, Pacific Grove, CA.

March 19-20, 1999: **American Chemical Society Shortcourse** "Crystallography for Chemists", CSU Fullerton

March 20, 1999: **Southern California Undergraduate Research Conference in Chemistry and Biochemistry, SCAURCON99**, CSU Fullerton

March 21-25, 1999: **American Chemical Society** National Meeting, Anaheim, CA.

May 22-26, 1999: **American Crystallographic Association** Annual Meeting, Buffalo, NY.

August 4-13, 1999: **International Union of Crystallography** Meeting, Glasgow, Scotland.

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